



Review

Nickel recovery/removal from industrial wastes: A review

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ABSTRACT

Nickel is an important metal, heavily utilized in industry mainly due to its anticorrosion properties. As a consequence, nickel containing wastes such as spent batteries and catalysts, wastewater and bleed-off electrolytes are generated in various processes. These wastes could have a negative impact on the environment and human health if they contaminate soil, water and air. The present review addresses the environmental and economical aspects of nickel recovery/removal from various types of wastes. The main physico-chemical technologies for processing various effluents and wastewaters containing nickel are reviewed and discussed. Nickel recovery from spent batteries, catalysts, electronic waste and other sources is described. Hydrometallurgical approaches are emphasized. Recovery of nickel from wastes is important not only for economical aspects, but also for environmental protection.

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1. Introduction

Nickel (Ni) a silver white, hard and ductile metal, is the 24th most abundant element in the Earth's crust having an estimated concentration of 0.008% (Kerfoot, 2005). Ni has an atomic number of 28 with electron configuration $1s^2 2s^2 2p^6 3s^2 3p^6 3d^8 4s^2$ and atomic mass of 58.71. It occurs in the periodic table in group 10,

following iron and cobalt, to which it is closely related, in the 4th period. Ni normally forms a face-centred cubic crystal lattice. In this configuration Ni is ferromagnetic at room temperature. A non-ferromagnetic hexagonal form of Ni is also known. Ni's physical and chemical properties, occurrence, extraction and purification methods are presented in detail in Ullmann's Encyclopedia of Industrial Chemistry (Kerfoot, 2005). The same source contains detailed information about Ni's compounds and alloys (Lascelles et al., 2005; Strassburg, 2005).

Ni has oxidation states from -1 to $+4$, but the $+2$ oxidation state is the most important. The oxidation of Ni(II) salt solutions

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with halides or persulfate produces the insoluble Ni(III) oxide (β -NiO(OH)), while the persulfate oxidation of Ni(II) hydroxide results in Ni(IV) oxide (NiO_2) (Kerfoot, 2005). Ni's most important quality consists in its ability when alloyed with other metals to increase the strength and corrosion resistance of the other metal over a wide temperature range (Strassburg, 2005).

Ni, Ni compounds and Ni alloys are used in many industrial and commercial applications. Most Ni is used for the production of stainless steel, non-ferrous alloys and Ni-based superalloys with high corrosion and temperature resistance properties (Reck et al., 2008). Ni alloys are widely used in areas ranging from industrial machineries to precision electronics. Some Ni compounds and complexes are used as efficient catalysts in various syntheses (Marafi and Stanislaus, 2008a, 2008b; Perosa and Tundo, 2005; Younkin et al., 2000). Ni oxide hydroxide is widely used in Ni-based rechargeable batteries (Shukla et al., 2001).

The extensive utilization of Ni-containing products sometimes leads to environmental pollution by Ni and its by-products (Denkhaus and Salnikow, 2002). Exposure to highly Ni-polluted environments has the potential to produce a variety of pathological effects in humans varying from contact dermatitis to lung fibrosis, cardiovascular and kidney diseases, and even cancer (Denkhaus and Salnikow, 2002; Kasprzak et al., 2003). Human exposure to Ni can originate from various sources (air, water, and food) (Cempel and Nikel, 2006). Since Ni is always present in the environment, the exposure to low amounts of Ni cannot be avoided and may not be harmful to humans (Denkhaus and Salnikow, 2002). Health problems arise due to exposure to high doses of Ni present in near or in Ni processing areas.

Ni has a relatively elevated price. The fluctuation in Ni price has a similar behaviour to many other metals (<http://www.infomine.com>). For example, between 1990 and 2004, the prices fluctuated between about 5 and 10 USD kg⁻¹, increasing in 2005 to about 17 USD kg⁻¹. In 2006, due to a sharp increase in demand (Reck et al., 2008), the price of Ni increased to more than 50 USD kg⁻¹ before declining to approximately 32 USD kg⁻¹. Recently, (June–July 2012) Ni price varied between 16 and 17 USD kg⁻¹.

With the above in mind, the recycling of Ni is an attractive option as it provides a means to recover and reuse the metal values and, at the same time, avoid environmental risks. The aim of the current review is to provide a survey of recent literature describing both Ni removal from wastewaters and recovery from various wastes including spent batteries, spent catalysts, bleed-off electrolytes and others.

2. Nickel removal from industrial wastewater

Due to the generation of large quantities of wastewater laden with potentially dangerous heavy metals, the electroplating industry poses a significant hazard to both the environment and human health. Different physico-chemical treatment techniques have emerged in the last decades, their advantages and limitations being evaluated in a number of recent and comprehensive review papers (Barakat, 2011; Chen, 2004; Fu and Wang, 2011; Kurniawan et al., 2006a; Zamboulis et al., 2011). The present study will only summarize the most often used techniques for Ni removal from wastewater. These techniques include precipitation, ion flotation, ion-exchange, membrane filtration, adsorption and several electrochemical treatment techniques including electro-coagulation, flotation, dialysis, deionization and deposition.

2.1. Chemical precipitation

Chemical precipitation is widely used for heavy metal removal from various effluents. Numerous literature examples are summarized in a recent review paper by Blais et al. (2008). Chemical

precipitation of Ni is achieved by raising the pH to basic conditions, typically pH 9–10, where the metal is transformed into the highly insoluble Ni hydroxide, Ni(OH)_2 . By using this technique, Giannopoulou and Panias (2008, 2007) have shown the possibility of separating Ni and Cu from acidic polymetallic aqueous solutions, by either electrodepositing Cu prior to Ni precipitation (Giannopoulou and Panias, 2007) or by precipitating Cu at lower pH values than Ni (Giannopoulou and Panias, 2008), with high removal rates for Ni of 99.65 and 99.76% respectively. The precipitation was performed at elevated temperatures, 65–95 °C, when Ni(OH)_2 could be obtained by supersaturation controlled precipitation (Sist and Demopoulos, 2003).

Another study has shown the possibility of obtaining Ni(OH)_2 by electrochemical precipitation in the presence of nitrate ions (Subbaiah et al., 2002). Using an electrolytic cell with a titanium anode and a stainless steel cathode, the authors show that Ni(OH)_2 could be obtained by using NiSO_4 and nitrate ions (nitric acid) in the solution. The authors observed that the current efficiency increases when elevating the concentration of Ni, but diminishes when the current density and temperature are increased.

2.2. Ion flotation

Ion flotation is a separation process by which non-surface active ions are separated from aqueous solutions by the addition of a surfactant (Doyle and Liu, 2003; Kurniawan et al., 2006a; Zamboulis et al., 2011). The ions to be removed are attached to the surfactant by either electrostatic or chelating interaction and are removed from the solution using a foam phase created by sparging a gas (air) into solution. In the case of Ni, sodium dodecylsulfate was used successfully as an anionic surfactant and the influence of a neutral chelating ligand, triethylenetetraamine (Trien), was studied (Doyle and Liu, 2003). Trien was shown to increase appreciably the removal rates for Cu^{2+} (~20%) and Ni^{2+} (~10%) during ion flotation with sodium dodecylsulfate. A similar study was conducted using a non-ionic surfactant, dodecyltriethylenetriamine (Ddien) (Liu and Doyle, 2009). The study revealed selective ion flotation behaviour of Ni^{2+} , Co^{2+} , and Cu^{2+} using Ddien. When at pH values around 9, Ni^{2+} and Co^{2+} ions were preferentially removed from the solution over Cu^{2+} ions. Ni^{2+} concentration was reduced from an initial value of 0.15 mM to 0.01 mM (93% reduction).

2.3. Ion-exchange

By using this technique, undesirable ions are replaced by other ions which are harmless to the environment (Dabrowski et al., 2004). Globally, ion exchange is one of the most frequently applied treatments for wastewaters loaded with heavy metals (Dabrowski et al., 2004; Kurniawan et al., 2006a). For Ni, various ion exchange resins and zeolites were shown to be efficient (Al-Haj Ali and El-Bishtawi, 1997; Alyüz and Veli, 2009; Argun, 2008; Dzyazko and Belyakov, 2004; Juang et al., 2003; Keane, 1998; Papadopoulos et al., 2004; Priya et al., 2009; Revathi et al., 2012). In some cases, ion-exchange is used in combination with other techniques in order to improve the removal efficiency of some ions from wastewater. For example, when using a combination of ion-exchange and precipitation processes, a higher reduction of Ni^{2+} concentration was obtained, from 94.2 to 98.3% (Papadopoulos et al., 2004). Another example for Ni^{2+} removal used a combination of ion-exchange and electrodialysis (Dzyazko and Belyakov, 2004).

2.4. Membrane filtration

Membrane filtration is used worldwide for heavy metals removal (Barakat, 2011; Fu and Wang, 2011; Kurniawan et al.,

Table 1Removal of Ni²⁺ from wastewater using membrane filtration.

Technique	Initial Ni ²⁺ concentration (mg/L)	Removal efficiency (%)	Operating pressure	Working pH	References
Micellar-enhanced UF	29.35	46–54	0.5–2	5.5	Akita et al. (1999)
	10	88–99.2	1–5	5–3–6.3	Yurlova et al. (2002)
	0.95–1.76	73.6–85.1	3	3.5–5	Landaburu-Aguirre et al. (2012)
Complexation – UF	50	98–99	2–4	3–9	Molinari et al. (2008)
	58.71	31.5–98.2	5	7–9	Borbély and Nagy (2009)
Chelation – UF	25	24.1–94.2	2–5	NA	Kryvoruchko et al. (2002)
Flotation – UF	3.3	98.5	<1	8–10	Blöcher et al. (2003)
RO	3.36	96.8	20	3.6–7	Qin et al. (2002)
	44–169	99.7	11	6.5–7.5	Ipek (2005)
	500	99.5	5	4–11	Mohsen-Nia et al. (2007)
NF	29.35–880.75	89–96	2.8	3–7	Ahn et al. (1999)
	5.87	92–93	2–8	4–8	Mohammad et al. (2004)
	5–250	92–98	4–20	2–8	Murthy and Chaudhari (2008)
	5–10	96.87–97.96	4–20	2–10	Murthy and Chaudhari (2009)

UF, ultrafiltration; RO, reverse osmosis; NF, nanofiltration; NA, not available.

2006a). Membrane filtration methods can be divided into ultrafiltration, reverse osmosis, and nanofiltration. Several examples of each method for Ni removal from wastewater are detailed below and summarized in Table 1.

Ultrafiltration (UF) uses membranes which allow the passage of water and low-molecular weight solutes, while retaining the macromolecules and hydrated metal ions which are larger than the pore size of the membrane. Sometimes the pore sizes of UF membranes (3–20 nm) are larger than dissolved metal ions in the form of hydrated ions (Fu and Wang, 2011). Since these ions would pass through the membrane, various hybrid methods have been proposed which combine the characteristics of two complementary techniques. In the case of Ni, micellar-enhanced ultrafiltration (Akita et al., 1999; Landaburu-Aguirre et al., 2012; Yurlova et al., 2002), complexation-ultrafiltration (Borbély and Nagy, 2009; Molinari et al., 2008), chelating-enhanced ultrafiltration (Kryvoruchko et al., 2002), and a combination of flotation (described above) and filtration (Blöcher et al., 2003) were tested. In the case of micellar-enhanced ultrafiltration, a surfactant is used to create micelles, while for complexation-ultrafiltration, complexation agents such as poly(ethylenimine) (PEI) and poly(acrylic acid) are used to specifically bind cations.

Reverse osmosis (RO) is a popular wastewater treatment option in chemical and environmental engineering (Fu and Wang, 2011). RO is a pressure driven membrane process whereby water can freely pass through a membrane while cationic compounds are retained (Kurniawan et al., 2006a). RO membrane pore sizes can be as small as 0.1 nm (Bohdziewicz et al., 1999). RO can be used for solutions with low amounts of dissolved metal (micromolar to millimolar range). For Ni removal, reverse osmosis has been shown to work effectively in a few studies (Ipek, 2005; Mohsen-Nia et al., 2007; Qin et al., 2002). Qin et al. suggest that a preliminary ultrafiltration step would decrease the fouling of the RO membranes and increase the efficiency of the removal process (Qin et al., 2002).

Nanofiltration (NF) has unique properties. The separation mechanism involves both steric (pore size) and electrical effects (negatively charged surface groups) (Kurniawan et al., 2006a; Van Der Bruggen and Vandecasteele, 2003). The membranes used for NF can be described as “low-pressure reverse osmosis membranes” (Van Der Bruggen and Vandecasteele, 2003). Various studies using nanofiltration demonstrate high rejection rate for Ni (>89%) (Ahn et al., 1999; Mohammad et al., 2004; Murthy and Chaudhari, 2009, 2008). The technique is applied at ambient temperatures in a large pH range and can be used for solutions with Ni concentrations in the millimolar range (Table 1).

2.5. Adsorption

Adsorption is commonly used as it affords low cost, ease of operation and simplicity of design (Barakat, 2011; Fu and Wang, 2011; Kurniawan et al., 2006a; Zamboulis et al., 2011). Adsorption is a mass transfer process by which a compound is transferred and accumulates at the interface between two phases (liquid–solid, gas–solid). The substance binds to the surface of the solid phase through physico/chemical interactions and becomes the adsorbate, while the solid on which adsorption takes place is called the adsorbent.

Many recent review papers describe and summarize technologies dealing with heavy metal adsorption from wastewater (Ahluwalia and Goyal, 2007; Arief et al., 2008; Babel and Kurniawan, 2003; Barakat, 2011; Bhatnagar and Sillanpää, 2010; Bhattacharyya and Gupta, 2008; Crini, 2005; Das, 2010; Farooq et al., 2010; Fu and Wang, 2011; Gavrilescu, 2004; Gupta et al., 2009; Kurniawan et al., 2006a,b; O'Connell et al., 2008; Sud et al., 2008; Wan Ngah and Hanafiah, 2008; Wang and Chen, 2009; Zamboulis et al., 2011). Among these reviews, some briefly discuss the adsorption amongst other physico-chemical wastewater treatment techniques (Barakat, 2011; Fu and Wang, 2011; Kurniawan et al., 2006a; Zamboulis et al., 2011), whereas others provide greatly, more specific detail and describe the use of low-cost adsorbents derived from agricultural waste, industrial by-products or natural materials (Arief et al., 2008; Babel and Kurniawan, 2003; Bhatnagar and Sillanpää, 2010; Bhattacharyya and Gupta, 2008; Crini, 2005; Gupta et al., 2009; Kurniawan et al., 2006b; O'Connell et al., 2008; Sud et al., 2008; Wan Ngah and Hanafiah, 2008). Other reviews deal with biosorption and different biosorbents used for heavy metal removal (Ahluwalia and Goyal, 2007; Arief et al., 2008; Farooq et al., 2010; Gavrilescu, 2004; Wang and Chen, 2009).

The aforementioned reviews contain numerous examples of adsorbents used for heavy metal removal, including Ni. Specifically for Ni²⁺ removal, many examples of adsorbents are given in the literature, varying from the use of zeolites (Mavrov et al., 2003; Rubio and Tessele, 1997), an *Escherichia coli* biofilm supported on zeolite (Quintelas et al., 2009), natural kaolinite (Gu and Evans, 2008; Jiang et al., 2010; Yavuz et al., 2003), multi-walled carbon nanotubes (Chen et al., 2009; Kandah and Meunier, 2007), chitosan coated PVC beads (Popuri et al., 2008), activated carbon prepared from apricot stone (Kobyas et al., 2005), olive stone waste (Fiol et al., 2006), grape stalks wastes (Villaescusa et al., 2004), tea factory waste (Malkoc and Nuhoglu, 2005), rice bran (Zafar et al., 2007) and orange peels (Ajmal et al., 2000). Most spent adsorbents can be regenerated using basic or acid solutions, and reused (Kurniawan et al., 2006b). The adsorption capacities of some recently described examples of

Table 2
Adsorbents used for Ni²⁺ removal from wastewaters.

Adsorbent	Adsorption capacity (mg/g)	References
Synthetic zeolite	60	Mavrov et al. (2003)
Natural kaolinite	2.79	Yavuz et al. (2003)
	5.3	Jiang et al. (2010)
MWCNTs	49.26	Kandah and Meunier (2007)
MWCNTs/iron oxide magnetic composites	9.18	Chen et al. (2009)
<i>Escherichia coli</i> biofilm	15	Quintelas et al. (2009)
Chitosan coated PVC beads	120.5	Popuri et al. (2008)
Apricot stone	27.21	Kobya et al. (2005)
Tea factory waste	15.26	Malkoc and Nuhoglu (2005)
Protonated rice bran	102	Zafar et al. (2007)
Orange peel	158	Ajmal et al. (2000)

MWCNTs, multi-walled carbon nanotubes.

adsorbents used for Ni²⁺ removal from aqueous solutions are given in Table 2.

2.6. Electrochemical treatment

Electrochemical technologies were used for wastewater treatment for more than a century (Chen, 2004). Ni removal from industrial process liquids by means of electrochemical techniques have been summarized previously (Koene and Janssen, 2001). In this summary, the main focus was the evaluation of electrolysis and electro dialysis. Below examples of more recent studies involving electrocoagulation, electroflotation, electro dialysis, electrodeionization, and electrodeposition are provided (see Table 3).

Electrocoagulation involves the generation of coagulants in situ by using consumable electrodes (Mollah et al., 2004). In this process, iron or aluminium anodes are dissolved by applying a potential to them, while hydrogen gas and hydroxyl ions are produced via water electrolysis at the cathode (Chen, 2004). Iron and aluminium will almost instantly become polymeric hydroxides, which are excellent coagulating agents (Mollah et al., 2004). In the case of aluminium, the metal ions are removed from the solution by several mechanisms: direct reduction at the cathode, hydroxides formation by the hydroxyl ions formed at the cathode, and co-precipitation with the aluminium hydroxides (Heidmann and Calmano, 2008). Another example using Ni is a study where the wastewater containing metals originating from Ni and zinc plating processes (concentration range, 230–280 mg/L), was successfully treated by electrocoagulation using stainless steel electrodes (Kabdaşlı et al., 2009). In this case, Ni and zinc were removed by hydroxide precipitation and incorporation in the colloidal material generated by the formation of Fe(OH)₃ flocs.

Electroflotation uses tiny bubbles of hydrogen and oxygen gases generated from water electrolysis in order to float pollutants up

to the surface of an effluent (Chen, 2004). Electroflotation can be used in combination with aluminium electrocoagulation. This was used for removal of heavy metals, including Ni, from polymetallic solutions (Belkacem et al., 2008).

Electrodialysis is a separation process in which ions are transported through ion-exchange membranes by the application of electricity between two electrodes. Tzanetakis et al. have studied the extraction of Ni and cobalt from their sulphate solutions using two cation exchange membranes (the perfluorosulfonic Nafion 117 and a new sulfonated polyvinylidene fluoride membrane), an anion exchange membrane (Neosepta AHA), a platinum oxide based coated titanium anode, and a stainless steel cathode (Tzanetakis et al., 2003). The temperature influence upon the amount of Ni extracted and current efficiency was evaluated. The authors demonstrated the feasibility of separating Ni from cobalt by employing an appropriate complexing agent (EDTA).

Electrodeionization combines ion-exchange and electro dialysis. In the case of Ni several examples have successfully applied this combination (Dermentzis, 2010; Dzyazko, 2006; Lu et al., 2010; Spoor et al., 2001a,b, 2002a,b,c,d). Using a cation-exchange resin bed placed between two ion-exchange membranes (an anion-selective membrane on the anode side and a cation-selective membrane on the cathode side) (Spoor et al., 2001a,b, 2002a), the authors demonstrated the possibility of a continuous process of Ni removal from a dilute solution (Spoor et al., 2002a,d). The Ni²⁺ ions were removed from the solution by the following mechanisms: transport through the solution phase to the cathode compartment, sorption in the ion-exchange resin and subsequent transport to the cathode compartment, or by precipitation as a hydroxide in the central compartment of the cell (Spoor et al., 2002a). Ni hydroxide formation in the ion-exchange compartment could be decreased by decreasing the process solution pH (Spoor et al., 2002b).

Electrodeposition can be applied for Ni removal/recovery from wastewaters when the concentration of the metal is high in solution (g/L). Therefore the mechanism and applications of this technology will be discussed in more detail in the Ni recovery section. A few examples of Ni removal/recovery from dilute solutions are given as well. Njau et al. showed that control of the process is very difficult, even when using 3D electrodes (Njau et al., 2000). Depending on the solution composition and electrolysis conditions, metallic Ni, Ni oxide and Ni hydroxide, as well as Ni oxyhydroxide, could be deposited. The study underlines the fact that electro dialysis can be used to preconcentrate Ni ions in solution (Njau et al., 2000). A promising result was obtained with initial Ni concentrations as low as 2 g/l by using rotating cathode metallic granules (5 mm in diameter) (Orhan et al., 2002). The authors showed that at 50 °C electrolyte temperature and 325 A/m² current density, current efficiency of 74% was attained, with an energy consumption of 4.2 kWh/kg Ni.

Table 3
Electrochemical treatment technologies for Ni²⁺ removal from wastewaters.

Method	Anode	Cathode	Initial Ni ²⁺ concentration (mg/l)	Removal efficiency	Electrical conditions	Initial pH	References
Electrocoagulation	Aluminium	Aluminium	50–250	NA	33 A/m ²	4.5–7.5	Heidmann and Calmano (2008)
	Stainless steel	Stainless steel	248–282	100%	90 A/m ²	6	
Electroflotation	Aluminium	Aluminium	100	99%	20 V	8	Belkacem et al. (2008)
Electrodialysis	Pt oxide coated Ti	Stainless steel	11.72	69%	400 A/m ²	NA	Tzanetakis et al. (2003)
Electrodeionization	Platinum	Platinum	4.76	40%	10 V	2–3	Dzyazko (2006)
	Platinized titanium	Graphite powder	100	100%	30 A/m ²	4	Dermentzis (2010)
	Platinum	Platinum	28–56	100%	5 V	2.65	Spoor et al. (2002d)
Electrodeposition	Graphite RDE	Platinum foil	27200	NA	100–500 A/m ²	3–4	Njau et al. (2000)
	Activated Ti	Metal granules	2000	90%	325 A/m ²	5.5	Orhan et al. (2002)

RDE, rotating disk electrode; NA, not available.

3. Nickel recovery from various wastes

Apart from solid wastes like spent batteries and catalysts, alloy scrap, some technological processes generate aqueous solutions with high amounts of Ni. When Ni is present in solution, depending on its concentration, the previously described physico-chemical techniques can be applied successfully. For solid wastes two distinct approaches can be utilized: (i) hydrometallurgy, where waste metal is leached into a solution and subsequently recovered from it; (ii) pyrometallurgy, where waste metal undergoes a thermal treatment in order to be recovered.

3.1. Nickel recovery from spent solutions

In the electro-refining step of copper anodes, a bleed off electrolyte is generated with a high content of Ni (up to 20 g/L) (Agrawal et al., 2008, 2007; Nyirenda and Phiri, 1998). Nyirenda and Phiri have shown that Ni removal from the electrolyte solution can be achieved either by precipitation with aqueous ammonia or by evaporative crystallization of Ni sulphate (Nyirenda and Phiri, 1998). A more recent approach demonstrated that a partial decopperisation, crystallization, solvent extraction and electrowinning route was suitable to produce pure copper and Ni powders (Agrawal et al., 2008, 2007). The Ni recovery rate was in some cases above 90% with a current efficiency of less than 50%.

Spent plating baths are another source of high amounts of dissolved Ni (Li et al., 1999; Tanaka et al., 2008; Vegliò et al., 2003). For these, several methods can be employed successfully: electro-dialysis (Li et al., 1999), electrowinning (Vegliò et al., 2003) and continuous solvent extraction (Tanaka et al., 2008).

3.2. Nickel recovery from spent batteries

Battery recycling is very important nowadays both for economical reasons, since they contain valuable materials which can be reused, and for environmental and human health protection purposes, as they can release many dangerous substances if disposed of inappropriately. Using recycled materials for battery manufacturing reduces energy requirements for primary material production compared to the extraction and purification from ores (Rydh and Karlström, 2002). Heavy metals are of great concern in this context. The potentially hazardous metals used in batteries are mercury, lead, cadmium, zinc, manganese and Ni (Bernardes et al., 2004). Several recent studies have summarized the processes and technologies involved in the recycling of various types of batteries (Bernardes et al., 2004; Espinosa et al., 2004; Sayilgan et al., 2009;

Xu et al., 2008). Since the aim of the current study is the recovery of Ni, the focus will be on batteries using Ni and its compounds for their construction.

Ni is currently used as the anode material mainly in Ni–cadmium (Ni–Cd) and Ni metal hydride (Ni–MH) batteries (Shukla et al., 2001). Mean Ni content in Ni–Cd batteries is 16.4% (Huang et al., 2009) and between 25 and 49% in Ni–MH batteries (Scott, 2009). These battery types are rechargeable and are categorized as the aqueous electrolytes type (Beck and Rüetschi, 2000). The Ni active material used is in fact Ni(III) oxide hydroxide (NiOOH), which is converted into Ni(II) hydroxide (Ni(OH)₂) during battery discharge and reformed during recharge. Since Cd proved to be a highly toxic metal, its replacement with a hydrogen-absorbing alloy in Ni–MH batteries became less environmentally problematic and, moreover, increased the performance of rechargeable batteries (Beck and Rüetschi, 2000; Fetcenko et al., 2007; Shukla et al., 2001).

Concerning battery-recycling, several approaches are currently used including separation of battery components followed by pyrometallurgical and/or hydrometallurgical technologies (Bernardes et al., 2004; Espinosa et al., 2004). The first separation step has limited applications but can lower the total cost of the process. Many pyrometallurgical processes, which recover materials by using elevated temperatures, are used industrially for the recycling of batteries. Hydrometallurgy uses acid or base leaching of the metals into a solution and the recovery of metals by the techniques described in detail in the Ni removal from wastewater section. Hydrometallurgy uses less energy compared to pyrometallurgy, but generates liquid wastes that need additional treatment.

In the following sections, several examples of processes presented in recent literature involving recycling of Ni–Cd, Ni–MH and some Li-ion batteries (see Table 4) will be discussed. The focus will be mainly on the hydrometallurgical processes since Espinosa et al. have excellently summarized a number of industrial pyrometallurgical processes used for recycling of batteries (Espinosa et al., 2004). Concerning Ni–Cd batteries, most pyrometallurgical processes perform the distillation of Cd using either an open furnace when Cd is recovered as a powder of cadmium oxide, or one with a closed, controlled atmosphere, where metallic Cd and high Ni-content alloy are obtained (Espinosa et al., 2004). A fundamental pyrometallurgical study was conducted by Espinosa and Tenório, by using coal as reducing agent to find the optimal conditions for Cd distillation and the main impurities present in recovered materials (Espinosa and Tenório, 2006). It was found that at temperatures below 900 °C the reactions involved in Cd recovery are too slow and that the main impurity found in the recovered Cd was Zn.

Table 4
Hydrometallurgical recovery of Ni from spent batteries.

Battery type	Acid leaching	Temperature (°C)	Time (h)	Recovery of other metals	Ni recovery form	R (%)	References
Ni–Cd	H ₂ SO ₄ + H ₂ O ₂	60	1	Cd electrowinning	NiCO ₃ precipitation	98	Bartolozzi et al. (1995)
	H ₂ SO ₄ , HNO ₃ , HCl, aqua regia	25–90	24	Cd electrowinning	Ni(OH) ₂ precipitation	97	Yang (2003)
	H ₂ SO ₄ + H ₂ O ₂	80	5	Cd electrowinning	Ni electrowinning	67	Rudnik and Nikiel (2007)
Ni–MH	H ₂ SO ₄	95	5	Rare earths and Co solvent extraction	Ni oxalate	96	Zhang et al. (1999)
	H ₂ SO ₄	90	4	Rare earths, Fe, Zn precipitation	Ni electrowinning	NA	Bertuol et al. (2009)
	H ₂ SO ₄	95	4	Rare earths, Fe, Zn, Mn, Co; solvent extraction	Ni(OH) ₂ precipitation	98	Li et al. (2009)
	H ₂ SO ₄ + H ₂ O ₂	30–70	1	Rare earths precipitation	Ni electrowinning	NA	Rodrigues and Mansur (2010)
	HCl	95	3	Rare earths and Co solvent extraction	Ni oxalate	96	Zhang et al. (1998)
	HCl	95	3	Rare earths precipitation	Ni, Ni–Co electrowinning	82	Tzanetakis and Scott (2004a,b)
				Co electrowinning			
Li-ion	H ₂ SO ₄ , HNO ₃	70	1	Co in solution	Ni in solution	95	Sakultung et al. (2008)
	H ₂ SO ₄ + H ₂ O ₂	NA	NA	Co solvent extraction, electrowinning	Ni electrowinning	NA	Lupi et al. (2005)

R: Recovery rate; NA: not available.

3.2.1. Nickel recovery of from spent Ni–Cd batteries

Most of the early reports dealing with the recycling of Ni–Cd batteries used mixed pyro- and hydrometallurgical technologies for the recovery of metals. Bartolozzi et al. provided an update on Ni–Cd battery recycling literature and proposed a complete hydrometallurgical approach where the electrode powder is separated from the solid metal supports and the external case is first leached with sulphuric acid and the resultant solution is electrolyzed for Cd galvanostatic deposition (Bartolozzi et al., 1995). In the final step Ni was recovered by precipitation as carbonate. The recovery rate was 98% of the total Ni content of the battery. The electrodeposited Cd had incorporated small amounts of Ni (1.2%) and the NiCO₃ had some Cd impurities (less than 0.5%). The Fe present in solution (metal support leaching) was precipitated prior to Cd electrodeposition. In similar working conditions, Yang used a potentiostatic electrodeposition approach for Cd, while Ni was precipitated as Ni(OH)₂ (Yang, 2003). In this case various concentrations of HCl, H₂SO₄, and HNO₃ were tested as leaching agents, with the best results obtained using 4 M HCl. A similar approach was adopted by Rudnik and Nikiel; the difference being the addition of H₂O₂ to the sulphuric acid leaching solution and the galvanostatic electrodeposition of both Cd and Ni (Rudnik and Nikiel, 2007). Once again Fe was precipitated as Fe(OH)₃. Although some Ni can be lost in the Fe precipitate, the authors emphasize the fact that Fe needs to be removed from the solution if pure Ni recovery is the objective. Efficient separation of Ni and Cd by electrodeposition from various simulated spent Ni–Cd battery solutions was achieved (Mayén-Mondragón et al., 2008). While nitrate was found to be unsuitable for Cd recovery, chloride and sulphate favoured Cd deposition and an efficient Ni–Cd separation.

An interesting approach for bringing the metals into solution is bioleaching. By using acid producing bacteria, several authors have shown the possibility of recovering the valuable materials from spent Ni–Cd batteries with high recovery rates (Cerruti et al., 1998; Zhao et al., 2008; Zhu et al., 2003). The resultant sludge, after neutralization with lime, met the environmental requirements for agricultural use (Zhu et al., 2003).

As mentioned before, once leached into solution, the metals from Ni–Cd batteries (Ni, Cd and Co) can be separated by solvent extraction (Nogueira and Delmas, 1999; Reddy and Priya, 2006). A high separation efficiency for Ni, Cd, and Co (>99%) can be obtained by controlling the concentration of the extracting agent and pH of the aqueous phase (Reddy and Priya, 2006).

3.2.2. Nickel recovery from spent Ni–MH batteries

Compared to Ni–Cd, Ni–MH batteries incorporate Co and an important amount of rare earths (RE). Cobalt addition to the hydrogen-absorbing alloy has been found to improve the cycling behaviour of Ni–MH cells (Shukla et al., 2001). Co content in a NiMH battery can vary between 2 to more than 20% (Råde and Andersson, 2001; Scott, 2009). A pyrometallurgical approach is not suitable due to rare earths losses in the slag (Lupi and Pilone, 2002; Scott, 2009) and moreover, a pyrometallurgical separation of Ni from Co is not feasible (Müller and Friedrich, 2006).

Several hydrometallurgical procedures are described in the literature for Ni–MH material recycling (Table 4). Of course, a mechanical pre-treatment is essential in order to separate the case (usually made of iron) from the electrodes and the electro-active material (Bertuol et al., 2006; Granata et al., 2012; Tenório and Espinosa, 2002). In the next stage, a magnetic separation of the polymeric fractions and the metallic ones can efficiently recover high amounts of Ni-based alloys (Bertuol et al., 2006; Tenório and Espinosa, 2002). The majority of the studies use acid leaching in order to bring the RE, Ni and Co into solution. The acids used were mostly either sulphuric (Bertuol et al., 2012, 2009; Innocenzi and Vegliò, 2012a,b; Li et al., 2009; Nan et al., 2006; Pietrelli et al.,

2002; Rabah et al., 2008; Rodrigues and Mansur, 2010; Zhang et al., 1999) or hydrochloric (Fernandes et al., 2013; Kanamori et al., 2009; Tzanetakis and Scott, 2004a,b; Zhang et al., 1998). The separation of REs from the solution can be achieved either by solvent extraction (Fernandes et al., 2013; Tzanetakis and Scott, 2004a,b; Zhang et al., 1999, 1998) or by selective precipitation at low pH (Bertuol et al., 2009; Fernandes et al., 2013; Innocenzi and Vegliò, 2012a; Nan et al., 2006; Pietrelli et al., 2002; Rodrigues and Mansur, 2010). Usually after this step a solution rich in Ni and Co is generated. Cobalt can be separated from Ni by solvent extraction using Cyanex 272 (Granata et al., 2012; Innocenzi and Vegliò, 2012b; Li et al., 2009; Nan et al., 2006; Zhang et al., 1999, 1998), and the two metals are recovered as their salts (e.g., oxalate, sulphate, hydroxide, etc.).

Rabah et al. has shown that prices of end products obtained by Ni–MH battery recycling were much lower compared to prices of the same chemicals prepared from primary resources (Rabah et al., 2008). Instead of separation, some studies evaluate the possibility of an electrochemical co-deposition of Co and Ni with high current efficiencies (Lupi and Pilone, 2002; Tzanetakis and Scott, 2004b). Tzanetakis and Scott observed that a desired composition and morphology of the deposit can be obtained by controlling the pH of the electrolyte and the applied current density (Tzanetakis and Scott, 2004b).

Apart from Ni–Cd and Ni–MH batteries, some authors have studied the possibility of recovering Ni and other valuable materials from lithium-ion batteries (Granata et al., 2012; Lupi and Pasquali, 2003; Lupi et al., 2005; Nan et al., 2006; Sakultung et al., 2008, 2007). Ni is used in these batteries in the form of a complex oxide, for example lithium Ni cobalt oxide or lithium Ni manganese cobalt oxide. In all the studies, the metals (Ni and Co) were brought into solution using acid leaching, and in the case of Lupi et al., Ni and Co were separated using solvent extraction (Cyanex 272) followed by electrowinning to recover Ni and Co (Lupi and Pasquali, 2003; Lupi et al., 2005).

3.3. Nickel recovery from spent catalysts

Ni compounds and complexes are used as efficient catalysts in various chemical processes (Marafi and Stanislaus, 2008a,b; Perosa and Tundo, 2005; Younkin et al., 2000). Numerous recent studies dealing with metal recovery from spent catalysts indicate the growing concerns about the fate of spent catalysts. Published results show that hydro- and pyrometallurgy are the most frequently used technologies for metal recovery from spent catalysts (Marafi and Stanislaus, 2008b; Singh, 2009).

When using a hydrometallurgical approach, Ni leaching from various sources can be performed using mineral acids or bases. The experimental conditions and the recovery rates for Ni from several recent papers are summarized in Table 5. Among the acids used, sulphuric acid is cheaper and Ni sulphate is commercially important due to its usage in electroplating (Al-Mansi and Abdel Monem, 2002). In this study the authors investigated the possibility of Ni recovery from a spent alumina based catalyst (NiO/Al₂O₃). By leaching NiO with sulphuric acid, Ni was recovered as Ni sulphate by direct crystallization and at the same time the possibility of reusing the alumina support was demonstrated. The conversion of Ni reached 99% when using a 50% acid concentration, a 1:12 solid liquid ratio, particle size below 500 microns and a 5 h contact time with constant stirring at 100 °C.

Another study used H₂SO₄ as a leaching agent for Ni recovery from a spent catalyst and showed that the dissolution of Ni is a diffusion controlled process. An increase in temperature and sulphuric acid concentration resulted in Ni recovery improvement (Sahu et al., 2005) with the metal being obtained as sulphide and oxalate.

Table 5
Hydrometallurgical recovery of Ni from spent catalysts.

Type of spent catalyst	Reagent	S/L ratio	Stirring rate (RPM)	Temperature (°C)	Time (h)	Ni recovery form	R (%)	References
NiO/Al ₂ O ₃	50% H ₂ SO ₄	1:12	800	100	5	Ni sulphate	99	Al-Mansi and Abdel Monem (2002)
NiO/Al ₂ O ₃ , SiO ₂	8% H ₂ SO ₄	1:10	NA	90	2	Ni oxalate	98	Sahu et al. (2005)
						Ni sulphide		
V, Ni, Fe, Si	1 M H ₂ SO ₄	1:5	200	80	0.5	Ni sulphate	96	Ognyanova et al. (2009)
NiO	67% H ₂ SO ₄	1:14	300	80	2.33	Ni sulphate	85	Idris et al. (2010)
Ni–Mo	1 M H ₂ SO ₄	1:10	200	80	4	Ni sulphate	96	Ferella et al. (2011)
NiO/Al ₂ O ₃	40% HNO ₃	1:10	Ultrasonic	90	0.83	Ni nitrate	95	Oza et al. (2011)
Mo, V, Ni/Al ₂ O ₃	10% citric acid	1:40	Ultrasonic	60	6	Ni citrate	95	Marafi and Stanislaus (2011)
NiO/Al ₂ O ₃	0.8 M EDTA	1:50	700	100	10	Ni sulphate	96.1	Goel et al. (2009)
Ni, CaO/Al ₂ O ₃	0.8 M EDTA	1:20	600	150	4	Ni sulphate	95	Vuyyuru et al. (2010)
						Ni nitrate		

R, Recovery rate; NA, not available.

Some studies also show that sulphuric acid is more efficient than hydrochloric acid for Ni recovery from spent catalyst (Idris et al., 2010). Another study showed that the leaching efficiency of some metals from spent catalysts can be improved using ultrasonic agitation (Marafi and Stanislaus, 2011; Oza et al., 2011). In the second example (Marafi and Stanislaus, 2011), an organic acid (citric acid) proved to be more efficient for leaching than sulphuric acid at a relatively low temperature (60 °C).

In some cases, alkaline solutions can also be used to selectively leach metals (Ognyanova et al., 2009). The researchers showed that vanadium can be extracted from a spent sulphuric acid catalyst by alkaline leaching. Since Ni is not dissolved by NaOH, a sequential acid leaching (H₂SO₄) can be conducted on the alkaline leaching residue to recover Ni.

Usually, physico-mechanical pre-processing of the waste is necessary in order to facilitate the leaching process (Al-Mansi and Abdel Monem, 2002; Ferella et al., 2011). A kinetic study of leaching of Ni from a spent catalyst in sulphuric acid performed for two particle sizes (coarse and fine) showed a two stage leaching process for the fine particles. The first step was diffusion controlled and the second governed by chemical reactions. A single step chemical reaction controlled mechanism was used for the coarse particles (Nazemi et al., 2011).

Another approach investigated for metal recovery from various sources involved integrated biological processes. For example, in the case of metal recovery from spent catalysts, bioleaching involving bacteria was successfully used (Beolchini et al., 2010; Bosio et al., 2008; Gholami et al., 2011). In the first case, iron/sulphur oxidizing bacteria (*Acidithiobacillus ferrooxidans*, *Acidithiobacillus thiooxidans* and *Leptospirillum ferrooxidans*) were used to extract Ni, vanadium and molybdenum (Beolchini et al., 2010). In the second example, Ni was recovered as Ni sulphide by leaching using *A. thiooxidans* and precipitation at room temperature with sulphide generated by *Desulfovibrio* sp. cultures (Bosio et al., 2008). When comparing the leaching efficacy of two different bacteria species, Gholami et al. obtained different results when using *A. ferrooxidans* versus *A. thiooxidans* (Gholami et al., 2011). With *A. ferrooxidans* the maximum extractions for Al, Co, Mo, and Ni were 63, 96, 84, and 99% respectively, while for *A. thiooxidans* the highest extraction degrees were 2.4, 83, 95 and 16% for the aforementioned metal series.

When pyrometallurgy is used the spent catalyst is sintered at high temperatures, for example at 1500 °C under plasma conditions (Wong et al., 2006). In this particular case (NiO/SiO₂ catalyst) NiO was reduced to Ni and the organic tar from the catalyst surface was decomposed and reduced to syngas (Co, CO₂ and H₂). The syngas may actually have been responsible for reduction of NiO to Ni.

Some recent studies tackle the possibility of using chelating agents, for example EDTA (ethylene diamine tetraacetic acid), for Ni recovery from spent catalysts (Goel et al., 2009; Vuyyuru et al., 2010). The dechelation is performed using mineral acids, such as

sulphuric acid (Goel et al., 2009; Vuyyuru et al., 2010), when NiSO₄ is obtained, or nitric acid (Vuyyuru et al., 2010), when Ni(NO₃)₂ is generated. The experimental details for both studies are summarized in Table 5.

Apart from the issues related to environmental protection where toxic waste generation is minimized, some recent studies showed that Ni recovery from spent catalysts can also be economically viable and profitable (Yang et al., 2011, 2010). The economic viability of the process depends greatly on the market value of Ni and the recovery technology costs, but the results of the aforementioned studies are encouraging from this perspective.

3.4. Nickel recovery from waste electrical and electronic equipment

Scientific and technological progress continually requires more advanced electrical and electronic equipment (EEE). As such EEE manufacturing has become one of the most important world activities but unfortunately it generates huge amounts of electronic waste. In the last decades the accumulation of waste electrical and electronic equipment (WEEE) has become a global problem (Babu et al., 2007; Robinson, 2009; Widmer et al., 2005). These wastes are a threat to the environment due to their high content of toxic materials. However, they are an important source of recyclable materials, especially valuable metals (e.g., Au, Ag, Pd, Cu, Ni, and Zn). Nowadays there are various approaches for the treatment and recycling of WEEE, involving pyro-, hydro- and bio-metallurgical processes (Cui and Zhang, 2008).

Ni is present in various amounts in WEEE depending on the type of waste. A recent study has shown that some metallic components of cathode ray tubes (electron gun, shadow mask) contain important amounts of Ni (25–45 wt.% of the total metal content) and Fe (50–70 wt.%), and small quantities of Mn, Co and Cr (Robotin et al., 2011). Printed circuit boards (PCBs) contain important amount of Ni. Between 1 and 3 wt.% Ni is found in PCBs from mobile phones and computers (Park and Fray, 2009; Veit et al., 2005; Yamane et al., 2011). In the first case, the recovery of Ni mainly involves the separation of iron from the solution obtained by acid leaching and Ni electrowinning (Robotin et al., 2011). PCBs have a more complex metal content and the separation/extraction processes have to deal with more metals, including Au, Ag, Cu, Sn, Pb, Zn, Fe, and Al (Park and Fray, 2009; Xiu and Zhang, 2009; Yamane et al., 2011). In this context, a mechanical processing step can concentrate the metals in a certain fraction using various methods such as size, magnetic, and electrostatic separation (Cui and Forssberg, 2003; Veit et al., 2005).

3.5. Other sources

Stainless steel production generates large amounts of waste which can be classified into slags and dusts. A recent review paper

summarizes the generation, composition, characteristics and the leaching behaviours of the wastes obtained from stainless steel processes (Huaiwei and Xin, 2011). An example involving the Ni present in the dust generated by stainless steel production in an electric arc furnace evaluated the possibility of metal recovery by manufacture of composite pellets with carbon and Fe–Si (Takano et al., 2005). The smelting–reduction experiments with these pellets presented a recovery yield of 90% for Ni when using Fe–Si.

Alloy scraps are another secondary source of Ni (Alex et al., 2001; Shen et al., 2008). Alex et al. have shown the possibility of obtaining pure Ni and cobalt salts by cupric chloride leaching of the scrap, solvent extraction and precipitation of the salts. The overall recovery by this process was almost 99% (Alex et al., 2001). Shen et al. used hydrochloric acid leaching at 95 °C for 3 h and obtained a leaching efficiency for Ni of 96% and an overall recovery of 95% by crystallizing Ni as NiCl (Shen et al., 2008).

4. Conclusions

This paper summarized removal and recovery of nickel as based on recent literature. The main physico-chemical technologies for heavy metal elimination from wastewater were presented and evaluated from the point of view of nickel removal. Nickel recovery from various types of wastes was described with emphasis on the hydrometallurgical approach. The high market price of nickel makes nickel recycling economically profitable. On the other hand, the environmental concerns and health risks related to nickel exposure impose the removal and recovery of nickel from wastes.

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